CZOCHRALSKI RUBY

SEMIANNUAL TECHNICAL SUMMARY REPORT

Period: 1 May 1964-31 December 1964

January 22, 1965

Contract Nonr-4132(00)
Program Code Number 3730
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Period: 1 May 1964-31 December 1964

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M.N. Plooster - Project Scientist
H.M. Dess - Group Leader
O.H. Nestor - Principal Investigator

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Union Carbide Corporation, Linde Division
Speedway Laboratories
P.O. Box 24184
Indianapolis, Indiana 46224

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ABSTRACT

The quality of Czochralski ruby was significantly improved during the report period. The gain resulted from closer controls on growth parameters, ambient atmosphere, and melt temperature.

Crystals grown under preferred conditions are bubble-free and of high optical quality. Optical paths through 6-mm-diameter x 4-5 cm-long x .03-.04 wt percent Cr_2O_3, 60° orientation rubies are uniform over the rod aperture to within a wavelength (CdI 6438A) or better. The passive beam divergence observed approaches the diffraction limit. 90° and 0° rubies grown on Verneuil seed rods are of lesser quality in that order. A rough estimate based on rocking-curve data indicates an 8-second upper limit on mosaic structure in current ruby. The remaining optical inhomogeneity is due principally to chromium variations and probably residual stress.

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CZOCHRALSKI RUBY

I. INTRODUCTION

The goal of Contract No. Nonr-4132(00) is "to develop the technique of growing sapphire uniformly doped with chromic oxide by the Czochralski method." Recognizing that this research program is geared to the stringent performance objectives of the ARPA/ONR Laser Program, it is clear that diffraction-limited laser material quality is desired.

The research effort has shown steady progress toward the above goal. The quality of ruby has been significantly improved over that described in the Annual Summary Report covering the first period of this contract ending April 30, 1964.[1)] It is considered to exceed that available from any other source and to be intrinsically good enough to satisfy immediate ARPA/ONR needs. Yet there is further improvement to be obtained and this is considered to be feasible within the confines of the Czochralski growth approach.

The improvement in ruby quality is documented in Section II where comparison is made against Verneuil ruby and against diffraction-limit criteria discussed below. Also included in Section II are results on specific defects contributing to the residual optical inhomogeneity. In Section III the follow-on approach is described.

Several quality objectives can be set down a priori in a quantitative way in this program. This is done here to provide a basis for measuring the quality of current Czochralski ruby against the diffraction-limit goal. That goal focuses attention not only on the uniformity of chromium doping but also on whether there are local variations in crystallographic orientation and residual stress within the ruby crystal and also on whether there are other defects that can act as light-scattering centers, such as gas bubbles or solid inclusions. In a practical sense "diffraction-limited" does not mean absolute uniformity with regard to chromium doping, orientation and stress level, nor absolute freedom from bubbles or inclusions. But it is certain that

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1) "Czochralski Ruby" Union Carbide Corporation, Linde Division Speedway Laboratories, July 8, 1964.

tolerances on these parameters are very stringent. At least with regard to doping, orientation, and stress uniformity, specific tolerances can be derived from the practical criterion that differences in optical paths parallel to the axis of the ruby rod should not exceed one-quarter wavelength. This criterion is the "Rayleigh limit" carrying the connotation that the maximum brightness (in Airy's disk) in the Fraunhofer diffraction pattern of a light beam traversing the ruby rod would be only about 20 percent less than that if the ruby were perfect.[2)]

The Rayleigh criterion is significantly less stringent than one assumed earlier in this program to calculate defect tolerances.[1)] The latter were recalculated based on the $\frac{\lambda}{4}$ limit. The new tolerance (center-to-edge variation) estimates, length-dependent but diameter-independent as implied by the Rayleigh criterion, are plotted versus rod length in Figure 1 for several factors affecting the refractive index of ruby.[3)] (The stress dependence of the refractive index is rather uncertain, so that the stress tolerance does not warrant close scrutiny.) For 8-inch- (20-cm) long rods desired in the present phase of the ARPA/ONR Laser Program, the tolerance estimates for diffraction-limited quality are as listed in Table I.

TABLE I. Diffraction-Limit (Rayleigh's) Tolerance Estimates for 8-Inch-Long Ruby Rods

Cr concentration	$\pm$ 0.0003 wt % Cr_2O_3
Orientation	
0°, 90° rods	35 arc-minutes
60° rods	0.4 arc-minutes
Residual stress	9 psi

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2) See "Applied Optics and Optical Design" by A. E. Conrady (Dover Publications, Inc., New York, 1960) Part Two, p. 626.

3) Based on refractive index dependencies summarized in Appendix I. Annual Summary Report dated July 8, 1964 under Contract Nonr-4132(00).

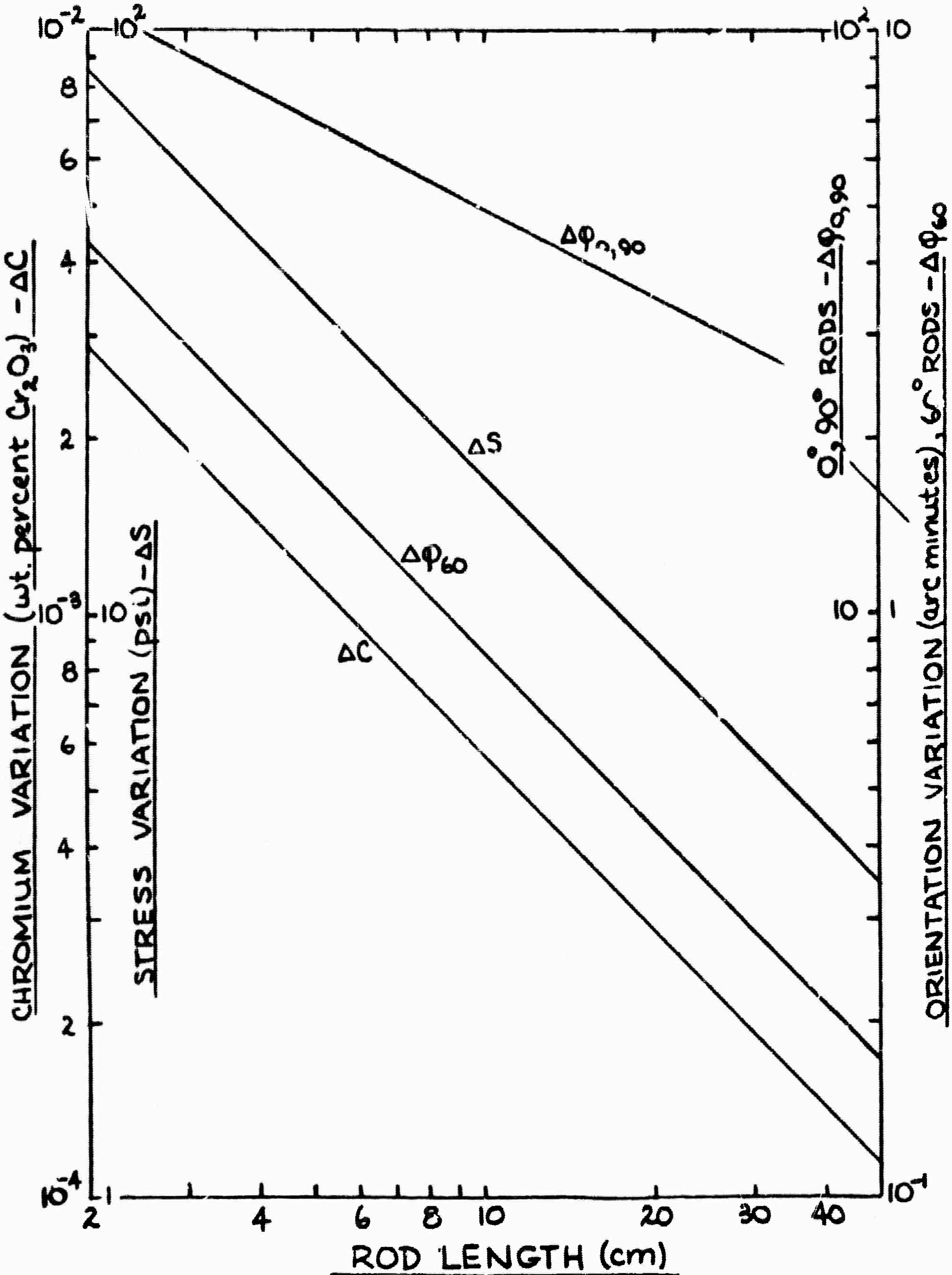

FIGURE 1. DEFECT TOLERANCES FOR $\lambda/4$ OPTICAL PATH DIFFERENCE

II. RESULTS

A. Growth Technique

The present apparatus represents a steady evolution from that used in growing the ruby product described in the First Annual Summary Report issued under this contract. Considerable effort was expended on modifying and improving the growth station to enhance control over experimental parameters. Improved atmosphere and temperature control were realized, leading to the significantly improved ruby described below.

The typical size of the boules grown in this research program is in the range 3/8-1/2 inch diameter x 2-3 inch length. Although the apparatus capability allows growth of larger boules, the above boule size yields large enough finished, cylindrical rods to identify the intrinsic capability of the technique. Rods were fabricated, generally without annealing, to dimensions typically 1/4 inch diameter x 1-1/2 to 2 inch length. Perhaps a further quality improvement could be realized by cutting samples of this size from a much larger boule. This has been avoided because it is, at least superficially, incongruous with economic, large-scale production practice, as is projected if the ARPA/ONR program shows technical feasibility.

Most of the boules have been grown on 60° Verneuil seed rods. All of the results below are for this orientation, except if noted otherwise. Only a few 90° and 0° crystals were grown. The 90° rods show some quality differences, generally below that of the 60° rods. 0° crystals are typically poorer than either the 60° or 90°, exhibiting gross misorientations apparently due to preferential propagation of defects in the C-axis direction. By contrast, 60° and 90° crystals even when viewed in the C-direction are of high quality.

B. Ruby Quality

The description of ruby quality in this section is based on the following tests:

1) Twyman-Green interferometry in which the spacing between interference fringes represents an optical path difference of $\lambda/2$. 6438A CdI radiation is used in this test.

2) High-angle-scattering-loss measurements in which a collimated beam of 7800A radiation, approximately 4 mm in diameter, passes down the ruby rod axis and the light lost through the cylindrical surface (ground finish) is measured via an integrating sphere. This component of the scattered radiation, expressed as a percentage of the incident radiation lost per unit length of crystal, is termed below the high-angle-scattering coefficient, without pretense that it is a total coefficient. The measurement scheme is shown in Figure 2.

3) Far-field photographs of a collimated beam (6328A) after passing through the ruby rod.

4) Far-field energy flux distribution measurements made with the apparatus sketched in Figure 3.

5) Measurement of chromium concentration variations in ruby windows via spectral absorption. Measurements were made on 2.5-mm-thick windows with an effective beam size of about 100 microns maximum dimension.

6) Schlieren examination for chrome banding

7) X-ray evaluation for subgrain structure

8) Visual inspection for bubbles or other "point-type" scattering defects under strong illumination and 15X magnification. Bubbles of size in the order of 1 micron are detectable by this method.

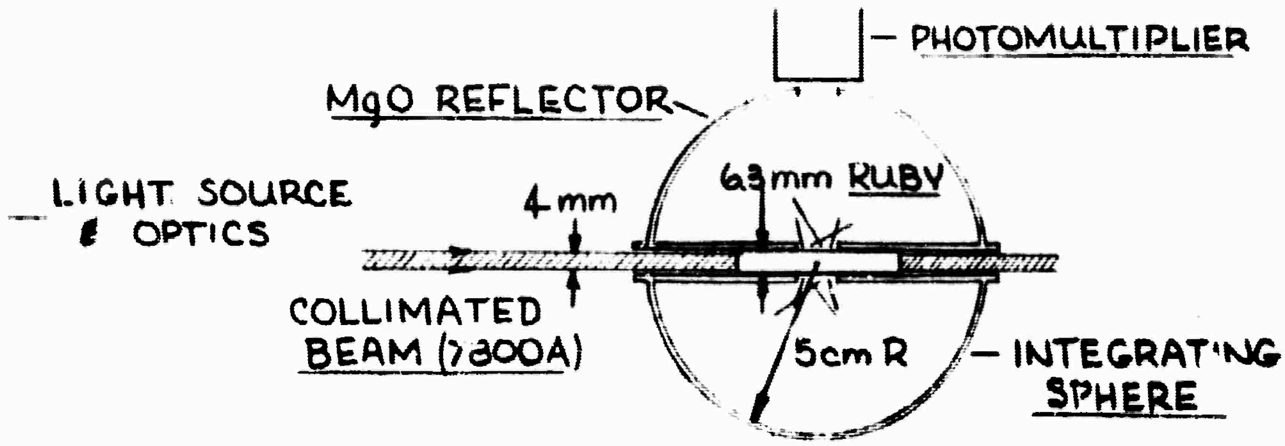

FIGURE 2. HIGH-ANGLE-SCATTERING MEASUREMENT SCHEME

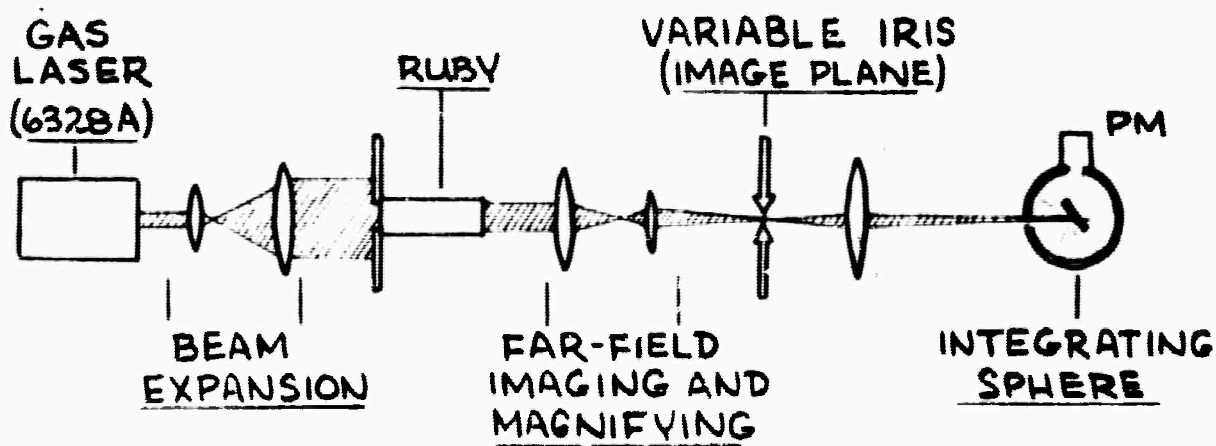

FIGURE 3. APPARATUS FOR MEASURING THE FAR-FIELD
ENERGY FLUX DISTRIBUTION

Twyman-Green and far-field photographs, far-field flux distribution measurements, chromium concentration determinations and Schlieren examinations were all made in polarized light with the E-vector normal to the C-axis of the ruby specimen.

1. Optical Homogeneity

The evolution of ruby quality is illustrated by the Twyman-Green interferograms, far-field (passive) photographs, and representative high-angle-scattering-loss coefficients shown in Figure 4. (The date "May 1964" identifies the beginning of the current contract period.) Included are three rods each of Verneuil ruby, Czochralski ruby as grown prior to the current contract period and present Czochralski ruby. The Verneuil rods exhibit variability beyond that found in the current Czochralski rubies, and generally are optically less uniform and accordingly distort a light beam to a greater extent. The "old" Czochralski product was characterized by optical inhomogeneities that were nearly axially-symmetric, as is evident in the central group of Twyman-Green interferograms. These were of significant magnitude, and distorted a light beam to a great extent. Nonetheless, the symmetry that prevailed indicated that a spectacular advance was available with finite effort. This is confirmed by the examples of current Czochralski rubies at the right of Figure 4. Optical path-lengths in the 4-5 cm-long specimens are consistently uniform to within a wavelength or better and the beam distortion effect is small enough so that in two of the far-field photographs the diameter of the Airy disk is discernible. Two of the current Czochralski specimens included in Figure 4 were cut from boules that were totally free of visible defects, such as bubbles; the third contained five "micro-bubbles." This is in sharp contrast to the high density of bubbles ($\gtrsim 10^3$ per cm^3) frequently evident in the Czochralski product before May 1964. Along with the above improvements it is noted that high-angle scattering has been reduced to a value below that of either the Verneuil or early Czochralski material. The central defect evident in the Twyman-Green interferograms of "new" Czochralski ruby are believed to be due to an observed increase in chromium concentration at that point, as will be discussed in a following section.

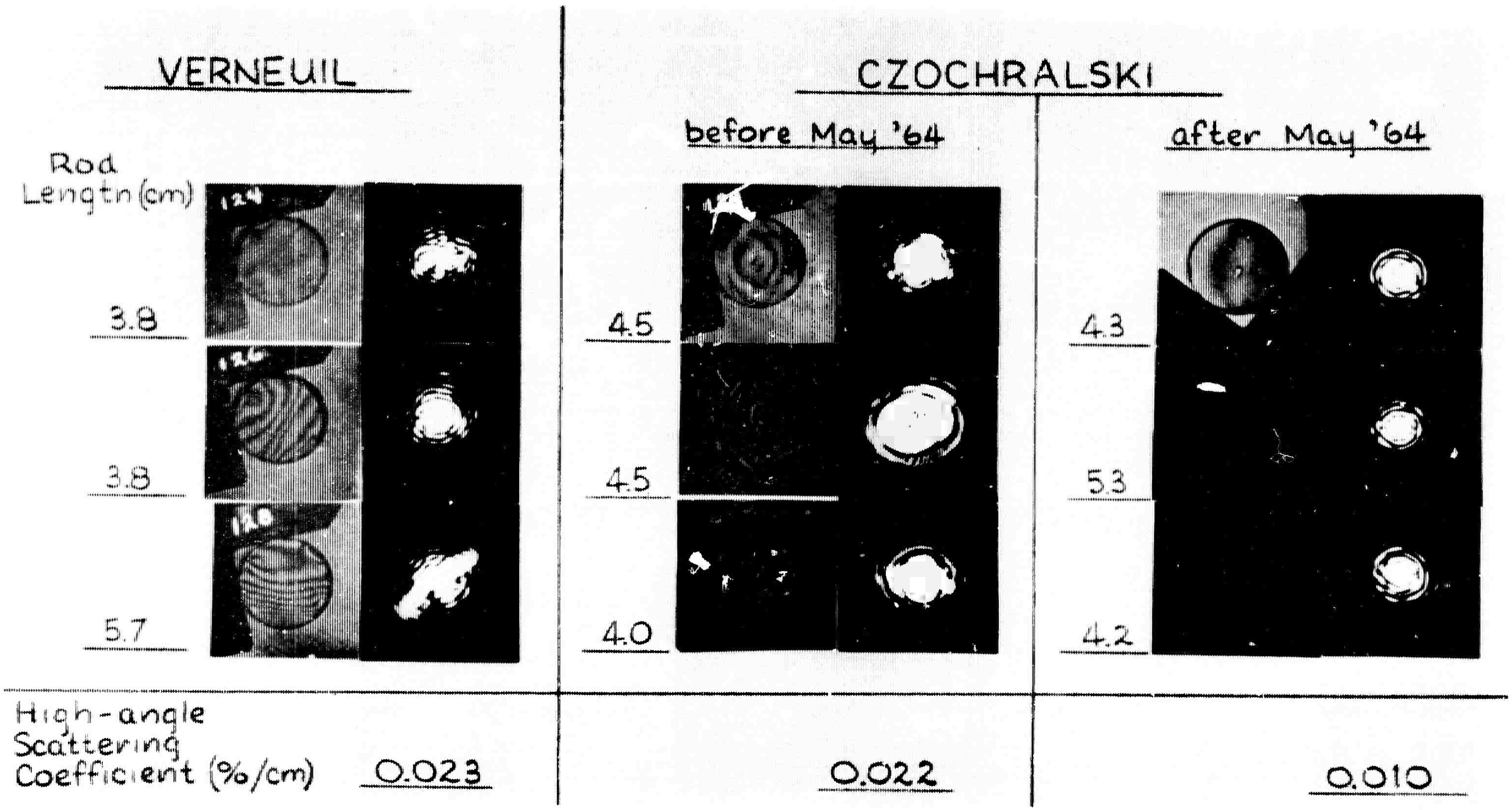

FIGURE 4. <u>THE EVOLUTION OF RUBY QUALITY</u>. The "old" Czochralski rod in the middle position is 5/16-inch diameter; the others are 1/4-inch (6.3 mm). Chromium content is nominally .03-.05 wt % Cr_2O_3. The probe beam was 5 mm diameter. The far-field pattern from the limiting aperture is shown at the right. All far-field photos are to the same magnification.

A quantitative appraisal of the flux distribution in the far-field pattern is shown in Figure 5 for Czochralski and Verneuil rubies of current quality in relation to the Fraunhofer limit. The 60° Czochralski rubies approach the theoretical limit quite closely and consistently, departing significantly therefrom only within Airy's disk defined by the first inflection point on the theoretical curve. It is apparent from this by comparison with the far-field photographs of Figure 4 that the latter, by virtue of limited latitude in film sensitivity, grossly obscure the quantitative characteristics of the far-field flux distribution. Figure 5 also illustrates the degradation of Czochralski ruby quality with crystal orientation in the order: 60°, 90°, 0°. Although results for two annealed Czochralski crystals are shown in Figure 5, too little has been done to draw firm conclusions on the effect of annealing. (Annealing is a potential avenue for quality refinement that, considering the stringent requirement on residual stress levels, can hardly be avoided in the ultimate approach to a diffraction-limited crystal.

Two of the 60° Verneuil rods included in Figure 5 are significantly inferior to the 60° Czochralski rods; the third Verneuil rod approaches the Czochralski rods. This variability, cited previously, is a characteristic of the Verneuil process that further distinguishes it from the Czochralski technique. The latter is subject to closer control and once optimum growth conditions have been established it has been shown to yield high quality consistently.

2. Chromium Variations

In Figure 6 chromium concentration profiles are plotted for five Czochralski ruby windows cut normal to the boule axis. Represented here are two 60° crystals (c and e) grown under conditions that are optimum for that orientation, a 0° crystal (a) and a 90° crystal (d) grown under the same conditions, and a 60° crystal grown under changed conditions. The following characteristics are noteworthy:

i) The radial chromium variations are nearly symmetric about the center, at least along the diameter that was traversed.

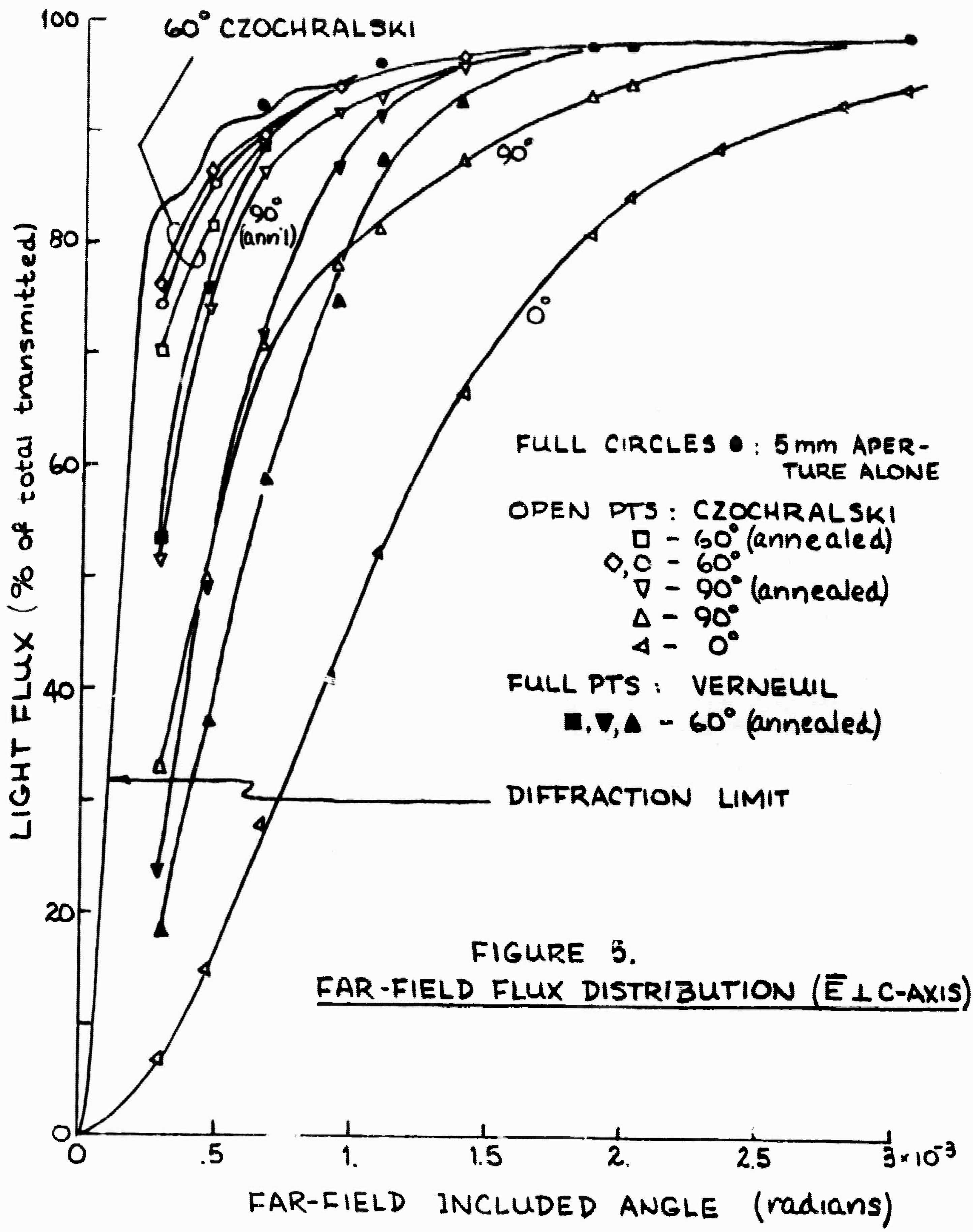

FIGURE 5. FAR-FIELD FLUX DISTRIBUTION ($\bar{E} \perp$ C-AXIS)

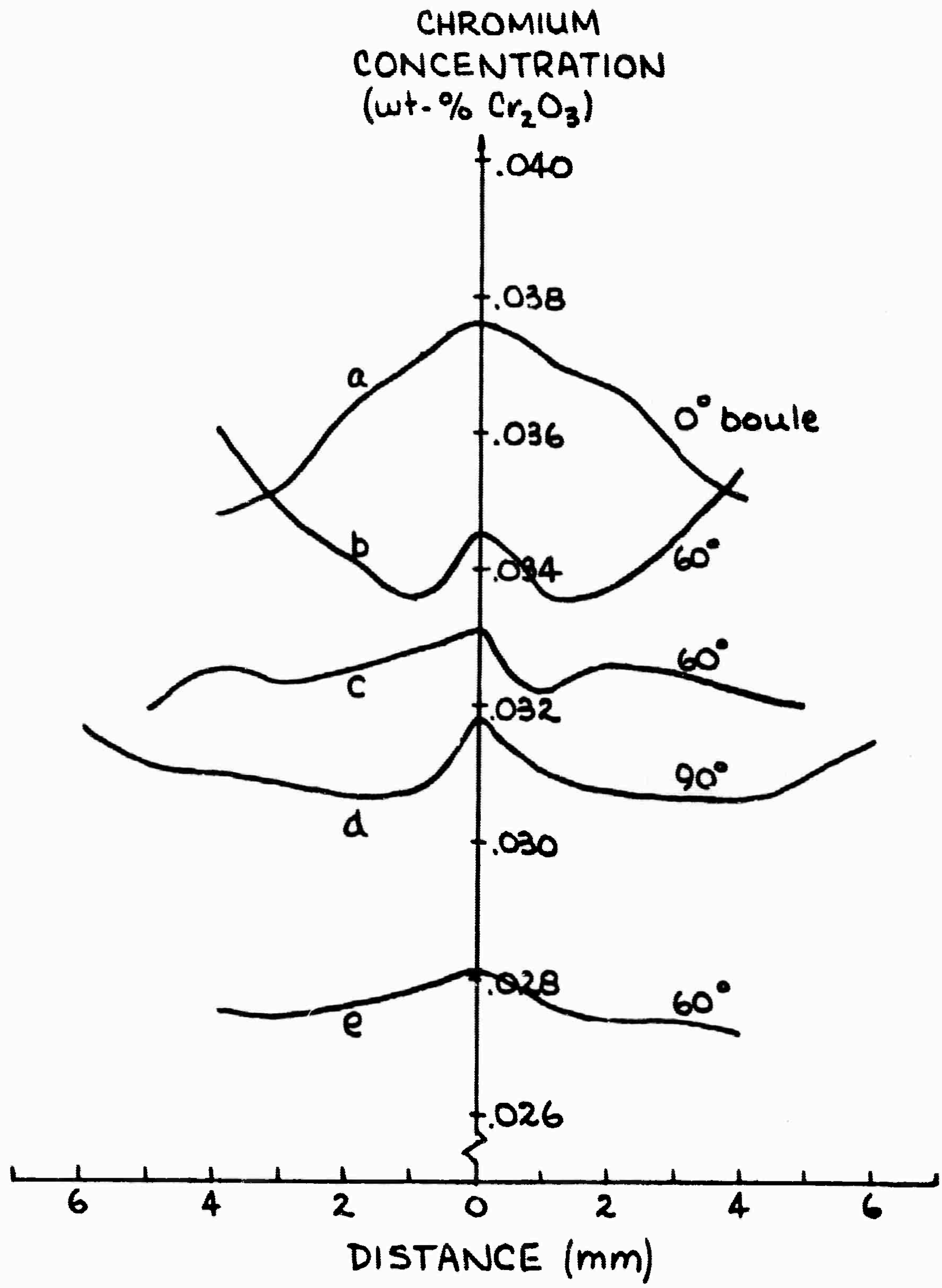

FIGURE 6. CHROMIUM CONCENTRATION PROFILES IN TRANSVERSE WINDOWS OF CZOCHRALSKI RUBY

ii) The magnitude of the variation lies in the range 0.001-0.003 wt percent Cr_2O_3. The variation for "optimum" 60°rods is about 0.001 wt percent Cr_2O_3. Assuming the variation in a rod is proportional to the average Cr concentration, a variation of 0.002-0.003 wt percent Cr_2O_3 is expected at the concentration levels of interest in the ARPA/ONR Program. Thus, to meet the 0.0003 wt percent Cr_2O_3 tolerance for diffraction-limited material, a tenfold improvement is needed and this must be preserved over the larger radius rods of interest in the Program.

If the Cr variations shown in Figure 6 represented the only defect and if these were uniform along the length of a 5-cm-long rod, Twyman-Green examination should reveal optical path differences of about $\frac{\lambda}{4} - \frac{3\lambda}{4}$ ($\frac{1}{2} - \frac{3}{2}$ fringes). This agrees favorably with what is observed. However more extensive measurements are needed (traversing different diameters for each of several windows cut from the same rod, correlating variations spatially with the Twyman-Green interferogram of the parent rod) before other causes of inhomogeneity can be ignored. Conversely it is clear that Cr uniformity needs attention.

iii) A common feature of the curves of Figure 6 is the more or less pronounced maximum in Cr concentration at the axis. This axially-localized Cr gradient is the only likely cause of the central defect evident in Twyman-Green interferograms (Figure 4) and in Schlieren photographs as well. Such defect has not been observed in limited growth trials of undoped sapphire, further supporting the hypothesis that they are associated with a Cr gradient. This phenomenon is thought to be related to a local change in effective segregation coefficient for Cr in the vicinity of the sharp pointed tip of the growth interface, perhaps dependent on hydrodynamic flow conditions past the interface and perhaps also on faceting that is observed at the tip. Figure 7 is a photograph of the tip of a 60° boule illustrating faceted growth. In almost all cases the facets appear to be the rhombohedral (r) planes of the sapphire lattice.

iv) In another aspect of Figure 6 it is seen that the direction of the radial gradient is not unique. There is an apparent effect of growth parameters (curve b versus curves c and e) that should be confirmed and delineated further.

FIGURE 7. THE TIP OF A 60° RUBY BOULE SHOWING FACETED GROWTH

Limited spectrophotometric determinations of the longitudinal Cr gradients in current Czochralski ruby give values in the range 2-4 percent (of the ambient concentration) per centimeter length. This agrees quite well with chemical analyses on sections taken from along a rod from which an overall gradient of 2.5 percent per centimeter was calculated. Since the crystal growth interface in this work is conical, a correlation between the sense of the radial and longitudinal chromium gradients should exist; in particular if the chromium concentration decreases with distance from the seed end, then it should decrease radially outward. In the samples examined thus far, this correspondence did not always hold. This apparent anomaly has not been resolved.

Local variations in Cr concentration were evident in "old" Czochralski ruby. These were pronounced enough to be easily discernible in shadowgraphs as shown at the top of Figure 8. With this inspection technique current rubies exhibit no banding. These became evident only in a Schlieren examination after considerable effort at improving the apparatus. Schlieren photographs of a one-centimeter cube sample cut from a 90° boule are shown at the bottom of Figure 8--one taken looking along the growth axis 0001 , displaying the central defect noted previously, and the others normal to the growth axis: $[01\bar{1}0]$ and $[2\bar{1}\bar{1}0]$. Striations matching the known contour of the growth interface are seen in the latter. It is assumed these represent Cr variations. A quantitative determination of the magnitude of the variations across the striae is not available. In the spectrophotometric work to measure longitudinal chromium gradients, fluctuations that might correlate with the above banding were not detectable, presumably because of limited sensitivity compounded by the use of thick (5-mm) samples.

3. Structural Evaluation

A one-centimeter cube of Czochralski ruby cut from a 90° boule (cited in the preceding paragraph) has been examined for the presence of low-angle misorientations by Dr. R.D. Deslattes of the National Bureau of Standards using a

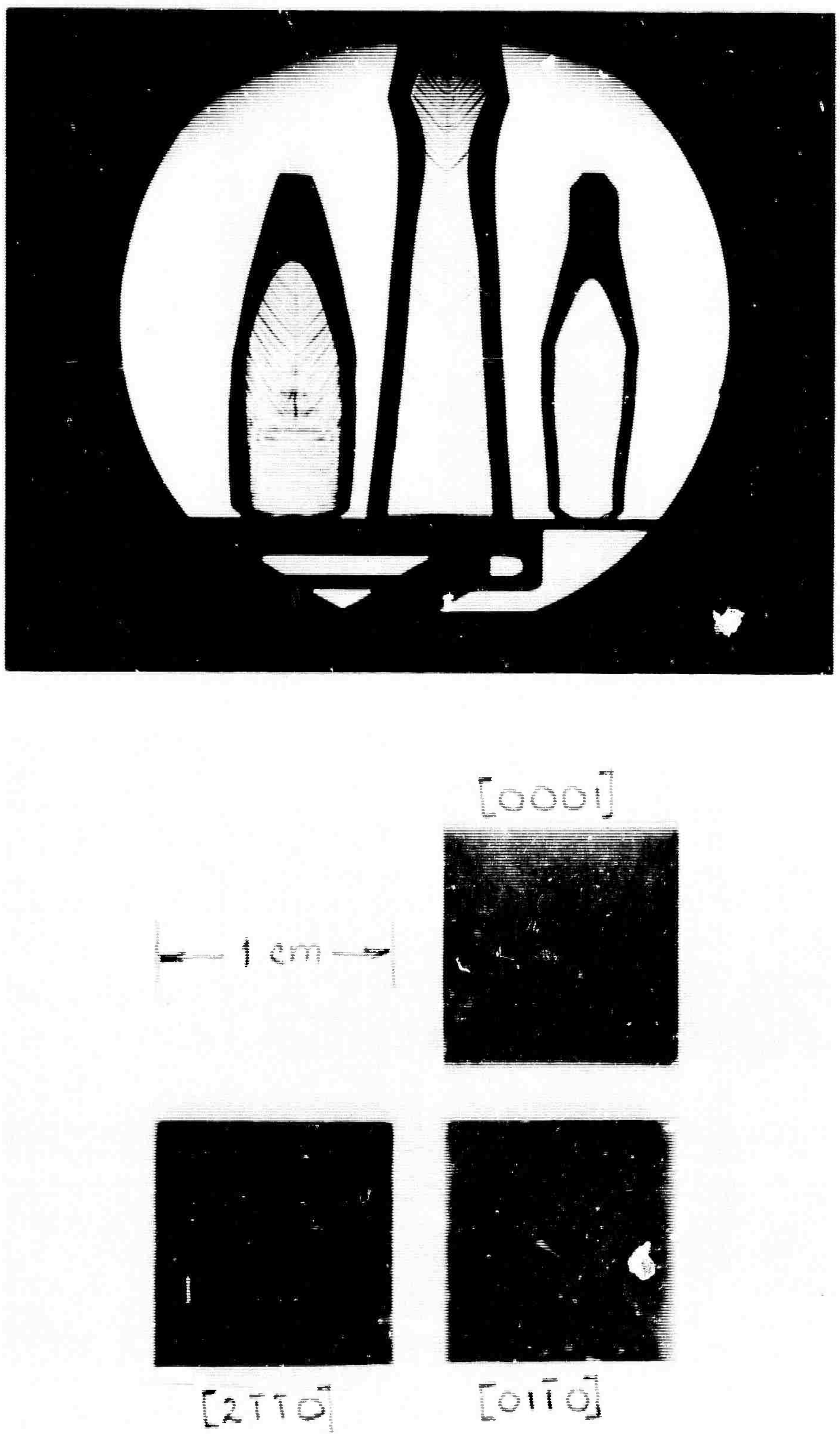

FIGURE 8. ILLUSTRATIONS OF CHROMIUM BANDING.
Top: shadowgraph of "old" Czochralski rubies.
Bottom: Schlieren photographs of a "new" Czochralski ruby cube viewed from different directions; growth direction [01$\bar{1}$0].

double-crystal X-ray spectrometer.[4] Dr. Deslattes observed a composite rocking curve at CuKα of about 16 arc-seconds full-width at half-maximum with respect to a first, or reference, crystal of dislocation-free silicon. The reflections operative were the (333) from silicon and the (000·12) from the ruby second crystal. The composite width included dispersive mismatch incurred by using different crystal compositions. Making rough allowance for this, the estimated combined effect of a cold-worked surface and subgrain boundaries was 8 arc-seconds. Even as an upper limit on misorientation this value is well within the tolerance listed in Table I. (The above cube, together with a second Czochralski specimen for rocking-curve studies, has been submitted to the Naval Research Laboratory for optical and structural evaluation, in accord with contract commitments.)

Although 60° rods exhibit no misorientations by the Schulz-Wei X-ray method practiced at the Speedway Laboratories (2 arc-minute detection limit), double-crystal X-ray measurements have not been made on such rods. On the basis of results cited in preceding sections it is to be expected that with 60° orientation, also, misorientations have been reduced to inconsequential status.

0° rods have many misorientation boundaries detectable by the Schulz-Wei technique.

4. Residual Stress

There are no quantitative data on the extent of residual stress in Czochralski ruby, except for an upper limit that could be derived from Twyman-Green interferograms considering stresses to be the only inhomogeneity present. Qualitatively, it has been evident since the early phases of this program that the stress level in as-grown Czochralski ruby is quite low. Qualitative results, however, do not suffice to rule out stress as a significant contributor to the remaining optical inhomogeneity in Czochralski ruby. In fact, limited sapphire growth experiments (that have not yet included optimum growth conditions) have not provided

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4) We gratefully acknowledge Dr. Deslattes' service and permission to cite this result.

material significantly better under Twyman-Green examination than current ruby. This suggests that the stress effect may well be comparable to the Cr-variation effect.

5. Bubbles

It has already been noted that crystals grown under the optimum growth conditions established in this program contain no or few visible bubbles, whether of macro- or micro-size, or similar light-scattering defects. The cumulative experience in this area convincingly demonstrates that control of such defects is a matter of proper growth conditions.

An additional contribution to "point" defects has been hypothesized to come from voids. The ease with which ruby crystals form r-plane facets (Figure 7) indicates that the ratio of growth rates along and normal to this plane is much greater than unity. Therefore, if for some reason a surface defect occurred during growth, an overgrowth of material along the r-plane could produce an elongated void of considerable length before growth normal to this direction could close the void. One elongated defect measuring 1μ x 100μ has been photographed.

III. FOLLOW-ON PROGRAM

A. Growth Studies

This phase of the program will concentrate on delineating the effects of system design, growth parameters and temperature control on ruby quality. Methods for controlling the growth interface shape will be explored, as this parameter is considered to exert itself on crystal quality. Better temperature control is a continuing objective to be pursued, not only through circuitry but also through system design; this problem area will be studied further. Special emphasis will be given to growing undoped sapphire to eliminate chromium variations as a cause of optical inhomogeneity and thereby to expose the effect of residual stresses.

B. Annealing Studies

New emphasis will be directed toward this area. The effect of annealing on both optical and crystallographic properties will be explored using undoped sapphire as the principal material for study. An optimum annealing schedule will be sought.

C. Crystal Evaluation

Longitudinal chromium gradients will be determined using an improved spectrophotometric technique. Electron microprobe measurements will be made to verify that the growth striae represent chromium variations and to determine the magnitude of concentration jumps. High-angle scattering and beam divergence measurements will be made on a series of rubies to establish a practical tolerance on bubble density. Fluorescence linewidths from current Czochralski rubies will be measured; correlation of results with overall optical quality will be examined. Lasing tests have been resumed with particular emphasis on measuring resonator losses.

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Westinghouse Electric Corporation
Research Laboratories
Pittsburgh 35, Pennsylvania

Prof. Donald S. McClure
Institute for the Study of Metals
University of Chicago
Chicago 37, Illinois

Dr. Daniel Grafstein
General Precision, Inc.
Aerospace Group
Little Falls, New Jersey

MORE ADDITIONS TO LIST 54

Professor R. C. Ohlmann
Westinghouse Research Laboratories
Pittsburgh, Pennsylvania

Dr. R. C. Linares
Perkin-Elmer Corporation
Solid State Materials Branch
Norwalk, Connecticut 06852

Dr. J. G. Atwood
Perkin-Elmer Corporation
Electro-Optical Division
Norwalk, Connecticut

Professor S. Claesson
Uppsala University
Uppsala, Sweden

FURTHER ADDITIONS TO LIST 54*

Robert L. Parker
National Bureau of Standards
Washington, D.C.

N.D. Schoenberger
Precision Instrument Company
3170 Porter Drive
Palo Alto, California

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